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6. AUTHOR(S)

M. Skowronski, Prof., Materials Science & Engineering
D. W. Greve, Prof., Electrical & Computer Engineering

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

Carnegie Mellon University
5000 Forbes Avenue
Pittsburgh, PA 15213-3890

8. PERFORMING ORGANIZATION REPORT NUMBER

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13. ABSTRACT *(Maximum 200 Words)*

Boron-gallium nitride, boron-aluminum nitride and gallium-aluminum nitride films have been deposited on sapphire substrates by organometallic vapor phase epitaxy and their properties have been investigated by Transmission Electron Microscopy, high resolution x-ray diffraction, and Hall effect. Solid solubility limits of boron in AlN and GaN have been determined to be 1 and 7%, respectively. Growth of $B_xGa_{1-x}N$ shows severe poisoning effect with growth rates dropping rapidly whenever sp^2-bonded BN phase is detected by x-ray diffraction. The quality of $B_xGa_{1-x}N$ and $B_xAl_{1-x}N$ films degrades with increasing x both in terms of changes of lattice parameter indicating random strains/composition variation and misorientations between low angle grains in the film. The concentration of point defects also increases resulting in increase of electron concentration at low x and deep center concentration at x>0.20. n-type doping of films with silicon allowed to achieve electron concentration in 10^{18} cm^{-3} range for x up to 0.55. For higher aluminum content silicon donors exhibit DX-like behavior.

14. SUBJECT TERMS

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Growth of Lattice Matched Nitride Alloys and Structures

Final Technical Report
AFOSR Grant F49629-95-1-0087
December 15, 1994-March 15, 1998

Marek Skowronski, D. W. Greve*
Department of Materials Science and Engineering
Carnegie Mellon University
Pittsburgh, PA 15213
***Department of Electrical and Computer Engineering**
Carnegie Mellon University
Pittsburgh, PA 15213

Research objectives

The primary objective of this grant was to study the deposition of ternary $Al_xB_{1-x}N$ and $Ga_xB_{1-x}N$ alloys and determination of their structural, optical , and electrical properties. Of particular interest were the compositions corresponding to lattice matching of boron-containing nitride alloys to silicon carbide substrates. Since we determined that the solid solubility of boron in Al and Ga-nitrides is very limited, in the second part of the project the focus was on deposition of $Al_xGa_{1-x}N$ films on sapphire. The material properties have been optimized for fabrication of UV solar blind photodetectors and in particular on doping and deep center $Al_xGa_{1-x}N$ films with high aluminum fraction. In addition, several issues in processing of nitrides were addressed.

Summary of research performed

A closely related project (AASERT addition to this contract entitled "Deposition of Gallium Nitride Epilayers by OMVPE", AFOSR Grant F49620-94-1-0392) focused on deposition of GaN films on sapphire substrates and investigations of their structural properties has been completed recently. The project had following accomplishments:

(i) GaN films on sapphire have been deposited and the type and density of extended defects have been determined. Dislocations were found to be arranged in the form of a cellular network with high dislocation density along the cell walls and almost dislocation free cell interiors. The misorientation between cell in the basal plane can be as high as several degrees in nominally high quality GaN.

(ii) New type of extended defects was observed in GaN films. They form a small diameter (5 - 30 nm) empty tubes propagating along the c-axis. The defects terminate on the film surface forming craters and frequently are associated with multiple screw and edge dislocations.

More detailed description of program goals ans accomplishments can be found in the final report of this project.

OMVPE growth of nitrides

An existing OMVPE system designed for GaAs growth has been modified in order to deposit high quality gallium and aluminum nitride films as well as their ternary solutions. Modifications included fitting in a second switching manifold designed to handle hydrides (in particular ammonia), replacing single inlet reactor by two inlet reactor, and upgrading the heating from infrared lamps to induction heating. All new hardware components are controlled by the upgraded software. The deposition system in the new configuration is capable of reaching temperatures up to 1250 °C and rapid heating and cooling. The high temperature capability is essential for growth optimization of high aluminum content AlGaN suitable for solar-blind UV detectors.

Most growth runs (unless specified otherwise) followed approach by the UCSB group. [Heying, 1996 #1346], [Keller, 1996 #1347] The growth was performed on basal plane sapphire substrates that were cleaned in organic solvents, etched in a mixture of hot H_3PO_4 and H_2SO_4 and heat treated in situ in hydrogen at 1100 °C for 10 min. prior to growth. Trimethylgallium, trimethylaluminum, triethylboron, and ammonia have been used as precursors with hydrogen as a carrier gas. At the

end of the annealing cycle, Al_2O_3 substrates were exposed to ammonia flow for approximately ten second and cooled in ammonia flow to 500 °C. At this point the trimethylgallium flow was directed to reactor and thin (nominal thickness 20 nm) GaN buffers were deposited. After buffer deposition, substrates were brought up to growth temperature of 1025 °C (unless otherwise stated) in ammonia flow and nitride films were grown with a growth rate of approximately 2 μm/h. Silane was used as a dopant source in layers intentionally doped n-type.

$B_xGa_{1-x}N/Al_2O_3$ deposition and properties

After establishing optimum growth conditions for GaN films, the $B_xGa_{1-x}N$ alloy growth experiments have been performed starting with addition of small amounts of triethylboron to the gas stream. The most striking result was rapid decrease of the growth rate at high temperatures (Fig. 1). For very small TEB flow rates, growth was not affected as compared to GaN, but with increased boron content (threshold value was dependent on temperature) the growth slowed down and at longer deposition times ceased.

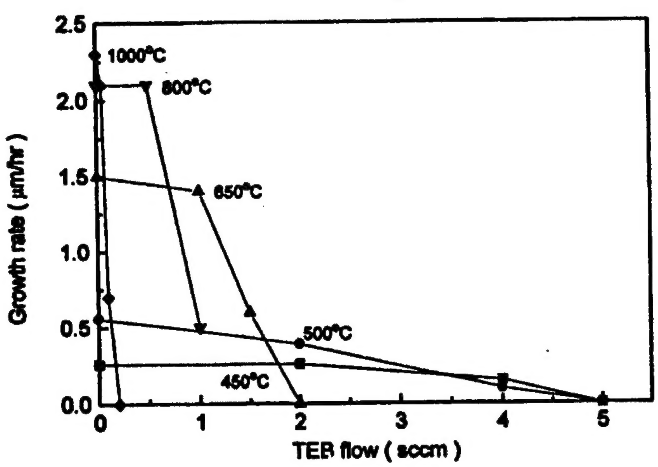

Fig. 1 The dependence of growth rate of $B_xGa_{1-x}N$ on TEB flow at different growth temperatures.

For small TEB flow rates below the threshold value, the x-ray diffraction data exhibited one peak only indicating single phase, single crystalline deposit. The position of the basal plane reflection for $B_xGa_{1-x}N$ films shifted with the increasing boron content (Fig. 2) which corresponds to decreasing lattice parameter with increasing boron content as expected from Veegard's law.

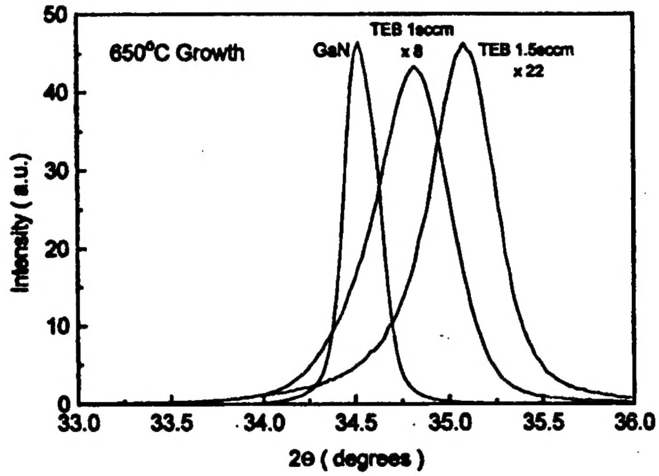

Fig. 2 Changes of position of (0002) reflection of $B_xGa_{1-x}N$ films.

At temperatures typical for high quality nitride film deposition to maximum amount of boron that was successfully dissolved was about one atomic percent. This amount increased at lower growth temperatures and reached maximum at 600 °C (Fig. 3).

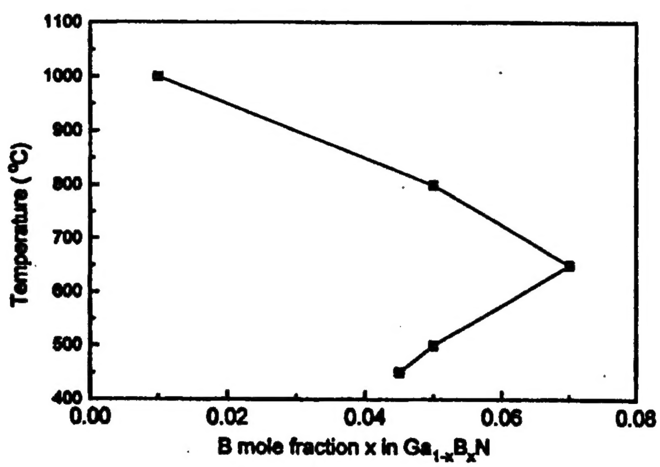

Fig. 3 Growth temperature dependence of the highest boron concentration attainable in $B_xGa_{1-x}N$ before the onset of growth poisoning.

This amount is still well below necessary boron content that would produce lattice matching to silicon carbide. In addition, the crystalline quality of the deposit at 600 °C degraded as measured by the width of x-ray diffraction peaks. Upon exceeding this limit additional x-ray diffraction peaks were observed (Fig. 4) and tentatively interpreted as due to nucleation of two additional

boron-rich phases: turbostratic BN and E-phase BN. Both of these phases are sp^2-bonded which can explain the drop of the growth rates. As soon as the sp^2-bonded phase of boron nitride formed on the film surface, the lack of broken bonds pointing upward resulted in much weaker bonding between BN layer and Ga adatoms. This resulted in re-evaporation of gallium. At the point when BN covered the entire surface, the growth ceased.

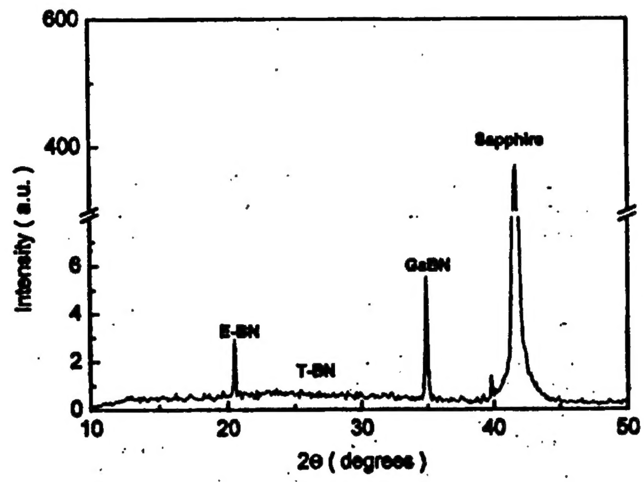

Fig. 4. X-ray diffraction pattern from two phase $B_xGa_{1-x}N$ film deposited at 650 °C.

The results summarized in the section are presented in detail in "Growth of GaBN ternary solutions by Organometallic Vapor Phase Epitaxy", A. Y. Polyakov, M. Shin, M. Skowronski, D. W. Greve, R. G. Wilson, A. V. Govorkov, R. M. Desrosiers, J. Electron. Materials **26**, 237 (1997).

$B_xAl_{1-x}N/Al_2O_3$ deposition and properties

A procedure similar to the one described above has been applied to deposition of $B_xAl_{1-x}N$ alloys. First the deposition conditions for the AlN films on sapphire have been optimized and this was followed by growth experiments with increasing flow of boron source. All growth runs have been performed at 1100 °C. Observed behavior was markedly different from the growth of $B_xGa_{1-x}N$ alloys. The growth rate slightly increased with the increased boron content. No growth poisoning was observed. Apparent interpretation of this fact was that even in the event of sp^2-bonded boron nitride phase nucleation of the layer surface, aluminum nitride was able to nucleate on BN due to much higher AlN bond strength which lowered the size of 2D critical nucleus. Boron content in $B_xAl_{1-x}N$ alloys was determined by SIMS technique in collaboration with Dr. R. G. Wilson at Hughes Research Laboratory. The boron content in all layers was proportional to the TEB flow and was as high as 40% in some layers. However, the x-ray diffraction peak for basal plane

reflection did not shift in accordance with Veegard's law. The largest value of the peaks shift corresponded to boron content in the film of about 1%. This behavior indicated the formation of a second boron-rich phase. It's nature was assessed by Transmission Electron Microscopy. Both plan-view and cross-sectional samples were investigated and results are presented in Fig. 5 and Fig. 6. Fig. 5 presents bright field image (cross section) of a structure consisting of two layers. The layer next to the sapphire substrate was AlN with the $B_xAl_{1-x}N$ layer of approximately equal thickness deposited on top. The AlN layer appears uniform in the image with clearly differentiated light and dark areas within the $B_xAl_{1-x}N$ alloy.

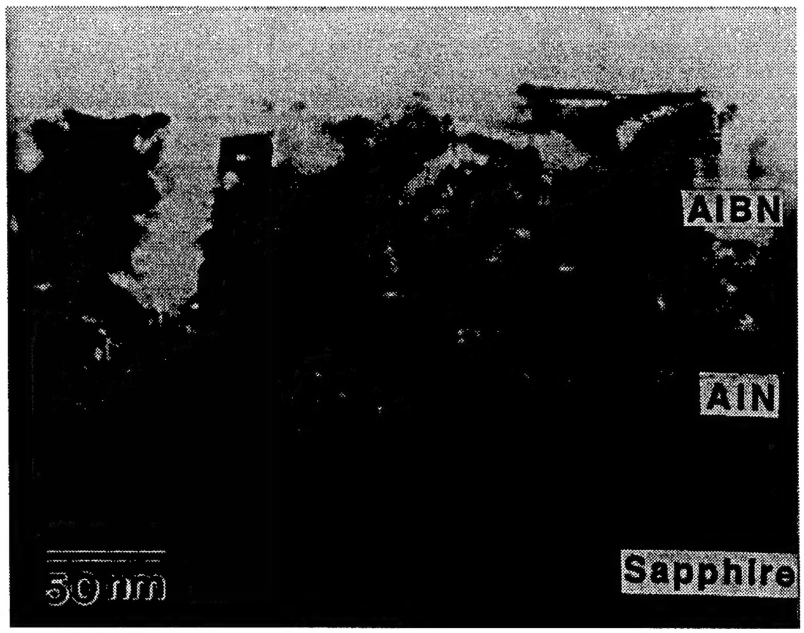

Fig. 5. Cross-sectional bright field image of structure consisting of $B_xAl_{1-x}N$ layer on top of AlN layer (on sapphire). Boron--rich precipitates appear lighter in color.

The selected area diffraction pattern collected on the same sample is shown in Fig. 6. The main pattern corresponds to single crystalline AlN film. Additional reflection (marked with the arrow) are due to boron-rich phase that's aligned along the c-axis with the AlN orientation. SAD pattern was used to determine the lattice constants of the BN phase and allowed to identify this as wurtzite boron nitride. This result is potentially interesting as the wurtzitic BN is sp^3-bonded and could be exhibit interesting semiconductor properties. The attempts to produce single phase wurtzite BN on AlN were not successful. Additional confirmation of the presence of wurtzite BN was obtained in x-ray diffraction. The difficulty of this experiment lies in the close proximity of the BN peak and sapphire. More intense sapphire reflection usually masks the boron nitride. Experiments

performed on sapphire covered with a thick AlN layer revealed presence of additional peak in x-ray pattern (Fig. 7) located at position corresponding to wurtzite BN.

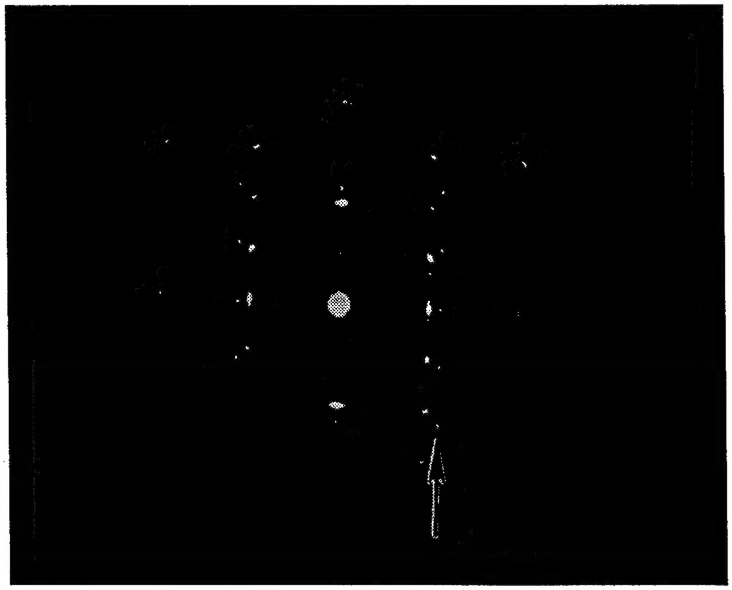

Fig. 6 SAD pattern for the cross section of $B_xAl_{1-x}N$ film. Additional reflections belonging to BN precipitates are marked with an arrow.

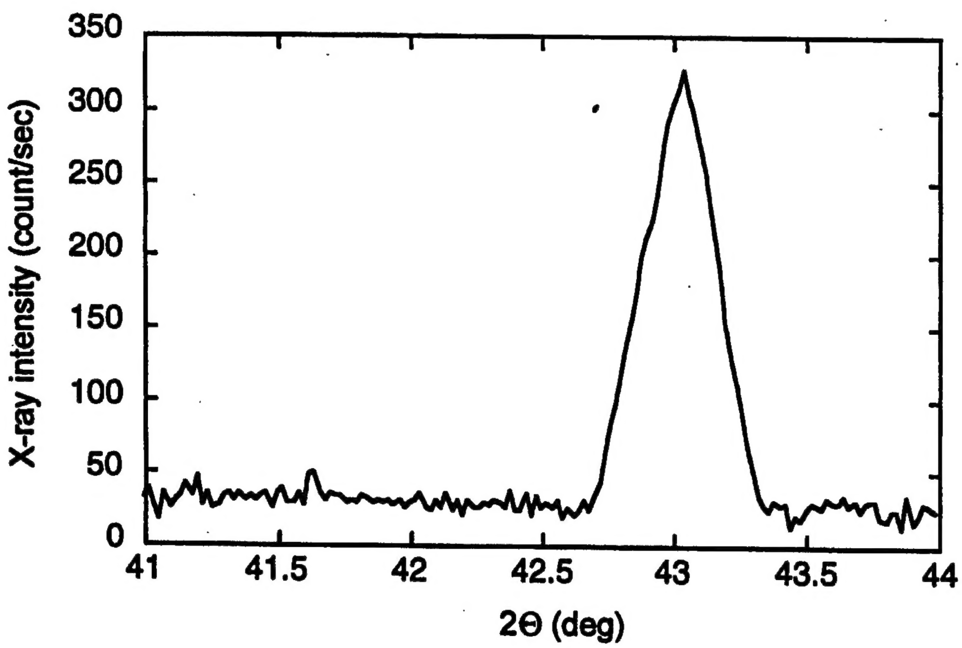

Fig. 7 X-ray diffraction peak for the thick $B_xAl_{1-x}N$/AlN sample showing w-BN peak at 43.04°.

The results summarized in this section are presented in detail in "Growth of AlBN solid solution by OMVPE", M. Shin, A. Y. Polyakov, W. Qian, M. Skowronski, D. W. Greve, and R. G. Wilson, Mat. Res. Soc. Symp. **449**, 141 (1997) and "Growth of AlBN solid solutions by organometallic vapor-phase epitaxy", A. Y. Polyakov, M. Shin, M. Skowronski, D. W. Greve, and R. G. Wilson, J. Appl. Phys. **81**, 1715 (1997).

$Al_xGa_{1-x}N$ deposition and properties

The structural quality of $Al_xGa_{1-x}N$ layers has been investigated as a function of aluminum content. A series of samples has been grown at precisely the same growth conditions with the only variable parameter being aluminum flow rate. The resulting films have been evaluated by high resolution x-ray diffraction using both basal plane and asymmetric reflections. Results for the 2θ-ω scans of (0002) reflection are shown in Fig. 8.

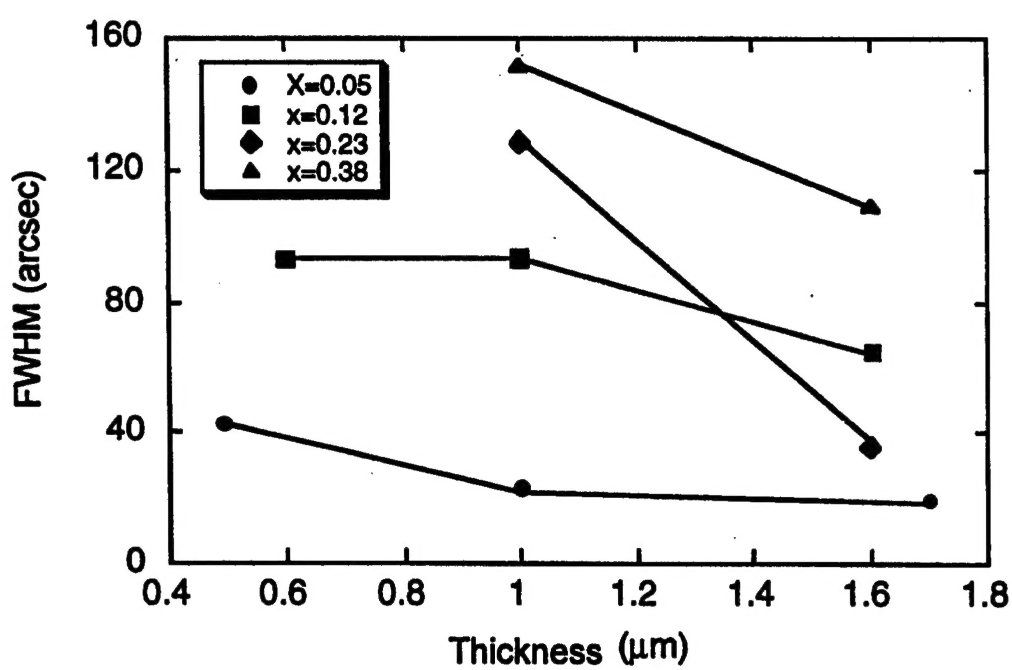

Fig. 8 Full width at half maximum of 2θ-ω scan of (0002) reflection from $Al_xGa_{1-x}N$ films deposited on sapphire.

2θ-ω scans reflect the amount of random strains and the composition variations along the c axis. Two trends are clearly visible. The first is the general increase of peak width with increasing aluminum content. For 1 μm thick layers, the width increases from 25 arc seconds for GaN films

to above 100 arc seconds for 30 % aluminum content. Secondly, the width decreases with increasing layer thickness. In GaN films this decrease stops after the layer thickens reaches about 1 micron which corresponds to coalescence of GaN islands and onset of layer-by-layer growth. It is clear, that the similar effect on $Al_xGa_{1-x}N$ films occurs at higher thicknesses and for the optimum layer quality deposits should have thicknesses in excess of two microns.

As determined in our previous work, nitride films are frequently composed of misoriented columnar grains that can be inclined to the c-axis or rotated about it. The degree of rotation is frequently much higher than the change in c-axis inclination and can be as large as several degrees. The width of w scan of (0002) reflection provides information about the degree of tilting in nitride film while the asymmetric reflection such as (10.5) used in this study is sensitive to both tilt and twist. Fig. 9 presents the data on full width at half maximum for both basal plane and asymmetric reflection as a function of aluminum content. As before width of reflections increase with increasing aluminum content. This indicates decreasing size of the columnar grains of $Al_xGa_{1-x}N$ caused by decreasing adatom mobility at constant growth temperature.

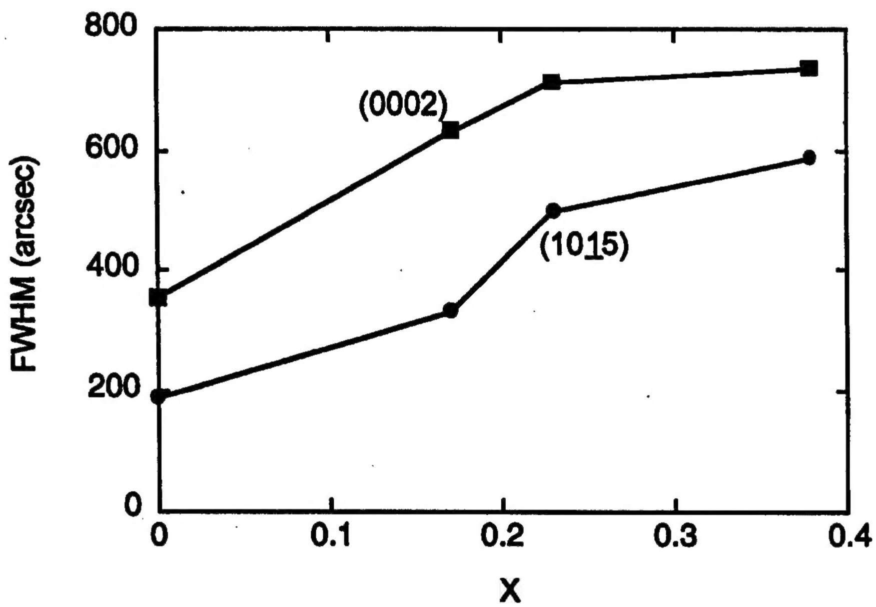

Fig. 9 Dependence of FWHM of ω-scan of (00.2) and (10.5) reflection on composition of $Al_xGa_{1-x}N$ films grown at 1025 °C.

Electrical properties of undoped $Al_xGa_{1-x}N$ films have been studied by Hall effect and low temperature photoluminescence measurements. Two series of samples have been grown at different deposition temperatures (1000 and 1050 °C). It was found that low growth temperatures

resulted in highly conductive material with electron concentrations in the 10^{18}-10^{19} cm^{-3} range. With increase of deposition temperature we were able to deposit n-type layers with electron concentrations below 10^{16} cm^{-3}. Both series of samples exhibited increasing resistivity in the high Al content range. Temperature dependent Hall effect measurements allowed to determine the Fermi level position (Fig. 10) versus composition. It was found that the centers pinning the Fermi level become increasing deep with increasing aluminum content. The nature of centers involved is not clear at present but they could involve oxygen either in the form of substitutional impurity or complex with other defects. The levels appear to follow the bottom of conduction band for low [Al] but above 30% they seem to be fixed to vacuum level.

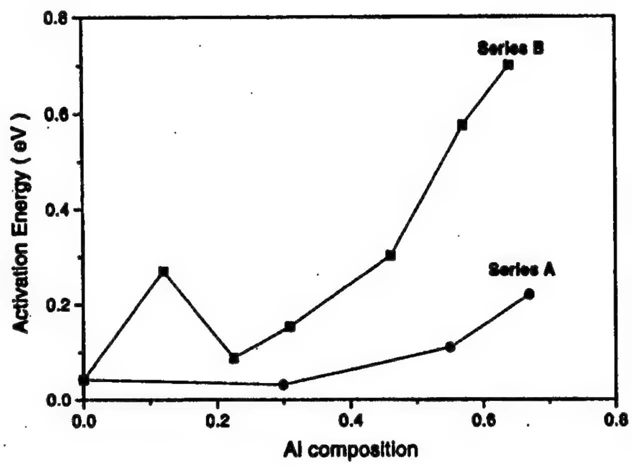

Fig. 10 Composition dependence of ionization energies of dominant donors in high temperature and low temperature $Al_xGa_{1-x}N$ series.

Controlled doping of high resistivity $Al_xGa_{1-x}N$ was investigated using silane. Electron concentrations in the $3x10^{18}$ cm^{-3} range have been achieved for aluminum fraction below 50% (Fig. 11) The decline of electron concentration at high aluminum content was determined to be due to increasing activation energy of silicon dopant. Similarly as in the case of donors in $Al_xGa_{1-x}As$, donors in AlGaN are expected to exhibit DX-like behavior i.e. for low x silicon should behave as a hydrogenic states with weakly bond electrons. At x>0.5, the ground state of a donor should by negatively charge center with donor atom occupying the interstitial position. Such a localized state has its energy fixed in respect to vacuum level and should emerge into the bandgap with increasing composition. The temperature dependent Hall effect measurements revealed the ionization energy of Si-donor rapidly increases between 50 and 60% aluminum (Fig. 12). In addition, the DX-type of behavior of silicon in $Al_xGa_{1-x}N$ was confirmed by hydrostatic pressure experiments performed

in collaboration with Unipress group and reported at the International Conference of Physics of Semiconductors in Jerusalem (1998).

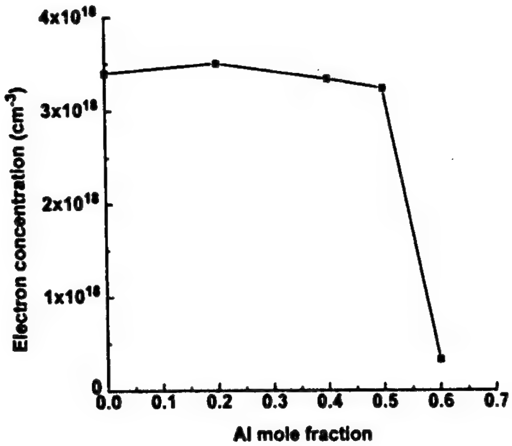

Fig. 11 Dependence of electron concentration on compositions of films doped with silane flow of 4 sccm.

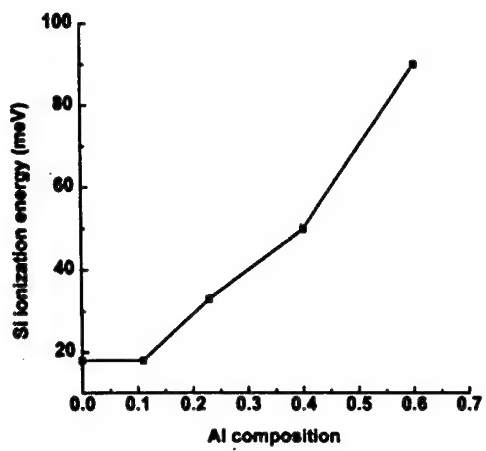

Fig. 12 Ionization energy of silicon donors in $Al_xGa_{1-x}N$.

AlGaN processing

Two different aspects of $Al_xGa_{1-x}N$ film post-growth processing has been investigated. The first has to do with improvement of optical properties of material intended for UV detectors. In the course of this project, it has been demonstrated that along with degradation of structural quality, the optical properties degrade as well. In particular, the slope of fundamental absorption edge decreases with increasing composition (Fig. 13). This is expected to cause increased signal in the

visible range of UV solar blind detectors and should be minimized. The effect was interpreted as a result of increasing point defect concentration (carbon and oxygen among others). The electric fields associated with charged centers broaden all optical transitions and produce characteristic Urbach edge. Fig. 13 shows the effect of hydrogenation on the tail of absorption edge.

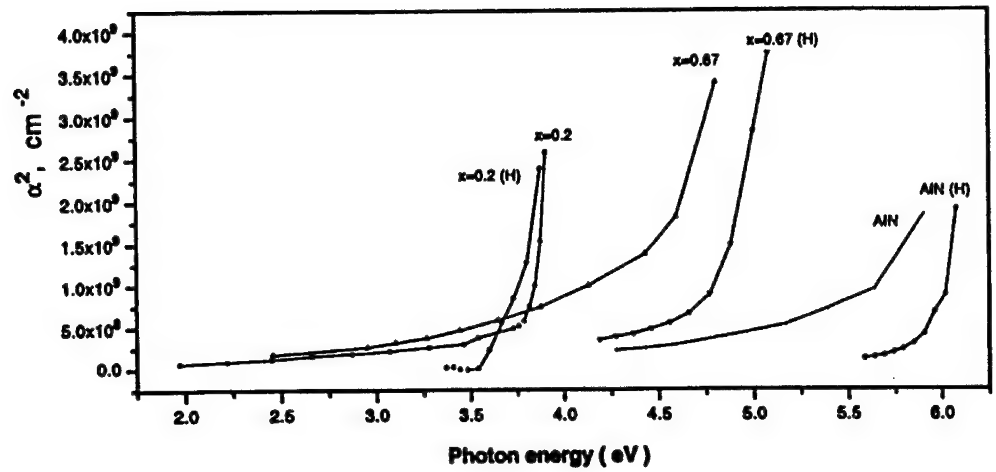

Fig. 13 Dependence of square of absorption coefficient on photon energy before and after hydrogen plasma treatment of $Al_xGa_{1-x}N$ films with various Al content. Spectra after hydrogen treatment are marked with H in brackets.

In all cases, the edge tail showed marked decease after hydrogenation. Hydrogen is known to form complexes with point defects frequently leading to formation of neutral complexes at the expense of charged centers. The decrease of total ionized defect content results in steeper absorption edge. An independent confirmation of this effect was obtained from the Hall effect measurements which showed an increase of electron mobility. Hydrogen-deep center complexes are thermally stable up to 550-700 oC depending on aluminum content.

In parallel, we have investigated selective doping of $Al_{0.12}Ga_{0.88}N$ films i.e. ion implantation. The films that were initially high resistivity were implanted with variety of ions including silicon, magnesium, and carbon. Only samples implanted with silicon showed significant increase of conductivity after post-implantation anneal at 800 °C in ammonia. This was the first report of successful implantation doping of $Al_xGa_{1-x}N$ material. The SIMS profiling showed negligible change in dopant distribution after anneal process for carbon and silicon but significant diffusion of magnesium.

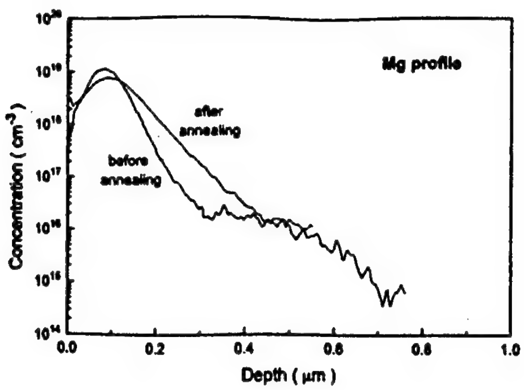

Fig. 14 SIMS profile of Mg in as implanted and annealed $Al_{0.12}Ga_{0.88}N$.

Polarity of nitride films

Significant research results have been obtained through collaboration with prof. R. M. Feenstra in Department of Physics, Carnegie Mellon University. Two types of polarities has been observed in MBE and MOCVD grown GaN films deposited on sapphire. In MBE growth, N-polar films are formed, while the MOCVD procedure outlined in the first section of this report result in Ga-polar surface ("Determination of wurtzite GaN lattice polarity based on surface reconstruction", A. R. Smith, R. M. Feenstra, D. W. Greve, M. S. Shin, M. Skowronski, J. Neugebauer, and J. E. Northrup, Appl. Phys. Lett. **72**, 2114 (1998); "Reconstructions of GaN(0001) and (0001) surfaces: Ga-rich metallic structures" J. Vac. Sci. Technol. B **16**, 2242 (1998); "Reconstructions of GaN(0001) and (0001) surfaces: Ga-rich metallic structures" A. R. Smith, R. M. Feenstra, D. W. Greve, M. S. Shin, M. Skowronski, J. Neugebauer, J. E. Northrup, J. Vac. Sci. Technol. B **16**, 2242 (1998)). The critical difference between two methods appear to be the first nucleation step in both growth procedures.

Publications resulting from project

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2. "Observation of nanopipes in α-GaN crystals", W. Qian, M. Skowronski, K. Doverspike, L. B. Rowland, and D. K. Gaskill, J. Crystal Growth **151**, 396 (1995)

3. "Open-core screw dislocations in GaN epilayers observed by scanning force microscopy and high-resolution transmission microscopy", W. Qian, G. S. Rohrer, M. Skowronski, K. Doverspike, L. B. Rowland, D. K. Gaskill, Appl. Phys. Lett. **67**, 2284 (1995)

4. "Structural defects and their relationship to nucleation of GaN thin films", W. Qian, M. Skowronski, and G. S. Rohrer, Mat. Res. Soc. Symp. Proc. **423**, 475 (1996)

5. "A microscopic evaluation of the surface structure of OMVPE deposited α-GaN epilayers", G. S. Rohrer, J. Payne, W. Qian, M. Skowronski, K. Doverspike, L. B. Rowland, and D. K. Gaskill, Mat. Res. Soc. Symp. Proc. **395**, 381 (1996)

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10. "High resistivity $Al_xGa_{1-x}N$ layers grown by MOCVD", A. Y. Polyakov, M. Shin, D. W. Greve, M. Skowronski, and R. G. Wilson, MRS Internet Journal, Nitride Semiconductor Research, **1**, art 36 (1996)

11. "Ion implantation of Si, Mg, and C into $Al_{0.12}Ga_{0.88}N$", A. Y. Polyakov, M. Shin, M. Skowronski, R. G. Wilson, D. W. Greve, and S. J. Pearton, Solid-State Electronics **41**, 703 (1997)

12. "Growth of AlBN solid solution by OMVPE", M. Shin, A. Y. Polyakov, W. Qian, M. Skowronski, D. W. Greve, and R. G. Wilson, Mat. Res. Soc. Symp. **449**, 141 (1997)

13. "High resolution x-ray diffraction analysis of GaN-based heterostructures grown by OMVPE", M. S. Goorsky, A. Y. Polyakov, M. Skowronski, M. Shin, and D. W. Greve, Mat. Res. Soc. Symp. **449**, 489 (1997)

14. "Studies of electrically and recombination active centers in undoped GaN grown by OMVPE", A. Y. Polyakov, A. V. Govorkov, N. B. Smirnov, M. Shin, M. Skowronski, and D. W. Greve, Mat. Res. Soc. Symp. **449**, 591 (1997)

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Personnel supported:

Faculty

M. Skowronski, Professor, Department of Materials Science and Engineering
D. W. Greve, Professor, Department of Electrical and computer Engineering
S. Mahajan, Professor, Department of Materials Science and Engineering

Graduate Students

Dr. M. Seub-Shin

(Ph. D. student, Department of Materials Science and Engineering, graduated in March 1998, thesis title: "Growth and Characterization of Group III Nitrides" (copy can be obtained from Science Library, Carnegie Mellon University, Pittsburgh PA 15213), accepted position with APA Optics, Blaine MN)

Seungook Min

(M. Sc. student, Department of Materials Science and Engineering, expected graduation date Spring 2000)

Postdoctoral Associates

Dr. A. Y. Polyakov, now at Inst. of Rare Metals, Moscow, Russia

Interactions and collaborations

Dr. D. K. Gaskill	Naval Research Laboratory, growth and TEM characterization of GaN microstructure
Dr. W. C. Mitchel	Air Force Research Laboratory, investigation of electrically active defects in AlGaN films
Maj. M. Estes	Air Force Research Laboratory, processing of UV solar blind photodetectors
Dr. K. Jones	Army Research Laboratory, investigation of AlGaN contamination by Secondary Ion Mass Spectroscopy
Dr. R. G. Wilson	Hughes Research Laboratories, investigation of impurities in GaN and AlGaN films
Dr. S. J. Pearton	University of Florida, dry etching of nitride films
Dr. H. Morkoc	University of Illinois, deposition of nitride films on ZnO
Dr. R. M. Feenstra	Carnegie Mellon University, nitride polarity and surface reconstructions
Dr. M. S. Goorsky	University of California, Los Angeles, x-ray characterization of nitride films
Dr. T. Suski	Unipress, Poland, behavior of Si donors under hydrostatic stress
Dr. A. Y. Polyakov	Institute of Rare Metals, Russia, investigation of deep centers in AlGaN films

most of the above collaborations resulted in joint publications which are preceding section.

Inventions/Patent Disclosures

None to date.